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IS 4335 (1985): 3-Chloroaniline [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]



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IS : 4335 - 1985

Indian Standard

SPECIFICATION FOR 3-CHLOROANILINE

(First Revision)

UDC 667.281 : 547.556.33'021



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MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

**AMENDMENT NO. 1 MARCH 2002
TO
IS 4335 : 1985 SPECIFICATION FOR
3-CHLOROANILINE**

(First Revision)

(*Page 3, foreword, clause 0.2, Structural formula*) — Insert '(CAS No. 108-42-9)' below the structural formula .

[*Page 4, Table 1, Sl No. (v), col 3*] — Substitute '99' for '98'.

(*Page 4, Table 1, Sl No. (vi), (d) 'Others'*] — Delete.

(PCD 11)

Reprography Unit, BIS, New Delhi, India

Indian Standard

SPECIFICATION FOR 3-CHLOROANILINE

(First Revision)

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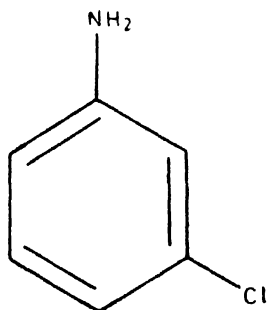
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Indian Standard
SPECIFICATION FOR 3-CHLOROANILINE
(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 10 January 1985, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 3-Chloroaniline (C_6H_5NCl) is widely used as a diazo component in dyestuffs of the azo series. It is also used in the manufacture of other intermediate dyestuffs and synthetic drugs. It has the following structural formula:



3-CHLOROANILINE
(Molecular Mass 127.57)

0.3 This standard was first published in 1967. The Committee responsible for the preparation of this standard decided to revise it in order to stipulate the requirement of maximum limits of impurities such as aniline, 2-chloroaniline, 4-chloroaniline and others. The requirement of distillation range has also been revised. Thin layer chromatographic test method has been incorporated in this version for estimation of impurities.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2 - 1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements methods of sampling and tests for 3-chloroaniline.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of colourless to pale reddish liquid free from visible impurities.

2.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR 3-CHLOROANILINE

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST, REF TO	
			Appendix A	IS : 5299 - 1969*
(1)	(2)	(3)	(4)	(5)
i)	Crystallizing point, °C, <i>Min</i>	-11	A-1	—
ii)	Distillation range	95 percent shall distill within 226·8 to 230·8 °C	—	6
iii)	Solubility in hydro- chloric acid	To pass the test	A-2	—
iv)	Moisture, percent by mass, <i>Max</i>	0·5	A-3	—
v)	Purity, percent by mass, <i>Min</i>	98	A-4	
vi)	Impurities, percent by mass, <i>Max</i> :		A-5	
	a) Aniline	0·5		
	b) 2-Chloroaniline	0·5		
	c) 4-Chloroaniline	0·5		
	d) Others	traces		

*Methods of sampling and tests for dye intermediates.

*Rules for rounding off numerical values (*revised*).

3. PACKING AND MARKING

3.1 Packing—Unless otherwise agreed to between the purchaser and the supplier the material shall be packed in suitable steel drums (*see* IS : 2552-1979*).

3.2 Marking — Each container shall be securely closed and shall bear legibly and indelibly the following information:

- a) Name of the material;
- b) Name of the manufacturer and his trade-mark, if any;
- c) Lot or batch number;
- d) Gross, net and tare mass; and
- e) Pictorial marking as per IS : 1260 (Part 1)-1973†.

3.2.1 Each container shall in addition, bear the minimum cautionary notice worded as under:

‘AVOID CONTACT WITH SKIN;
DO NOT HEAT TO DECOMPOSITION’

3.2.2 Each container may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions, under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in Appendix B.

5. TEST METHODS

5.1 Tests shall be conducted according to the methods prescribed in Appendix A and IS : 5299-1969‡. References to relevant clauses are given in col 4 and 5 of Table 1.

*Specification for steel drums (galvanized and ungalvanized) (*second revision*).

†Pictorial markings for handling and labelling of goods : Part 1 Dangerous goods (*first revision*).

‡Methods of sampling and tests for dye intermediates.

5.2 Quality of Reagents — Unless otherwise specified, pure chemicals and distilled water (see IS: 1070-1977*) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

(Table 1 and Clause 5.1)

METHODS OF TEST FOR 3-CHLOROANILINE

A-1. DETERMINATION OF CRYSTALLIZING POINT

A-1.1 Apparatus — The crystallizing point apparatus of the shape, dimensions and tolerance given in Fig. 1 consists of the following:

A-1.1.1 Outer Glass Test Tube — Serves as an air-jacket and is weighed with lead shots or similar loading material. It is provided with a cork through which the inner tube (A-1.1.2) is held in position.

A-1.1.2 Inner Glass Test Tube — Fitted with a cork which carries a stirrer in the form of a loop of glass with a glass stem and the thermometer placed centrally within the tube and the glass loop. The bottom of the bulb of the thermometer shall be about 10 mm from the bottom of the inner tube. The cork is so fixed that the immersion mark on the thermometer is in level with the top of the cork.

A-1.1.3 Cooling Bath — 1 000-ml glass beaker about 150 mm in height. The level of the cooling liquid in the bath shall be at least as high as the level of the sample in the inner tube.

A-1.1.4 Thermometer† — Conforming to the following requirements:

Range	—20° to +10°C
Immersion	76 mm
Graduation	at each 0.1°C
Long lines	at each 0.5°C
Number	at each 1.0°C
Scale error, Max	0.1°C
Expansion chamber	Permit heating to 60°C
Total length	370 ± 5 mm

*Specification for water for general laboratory use (second revision).

†Thermometers with the American Society for Testing and Materials, Designation 89C-59T, conform to these requirements.

Stem diameter	6.0 to 7.0 mm
Bulb length	18 to 28 mm
Bulb diameter	Not greater than stem and not less than 5.0 mm
Distance from bottom of bulb to bottom of scale	116 to 130 mm
Distance from bottom of bulb to top of scale	315 to 335 mm

Any thermometer of similar range and accuracy may be used.

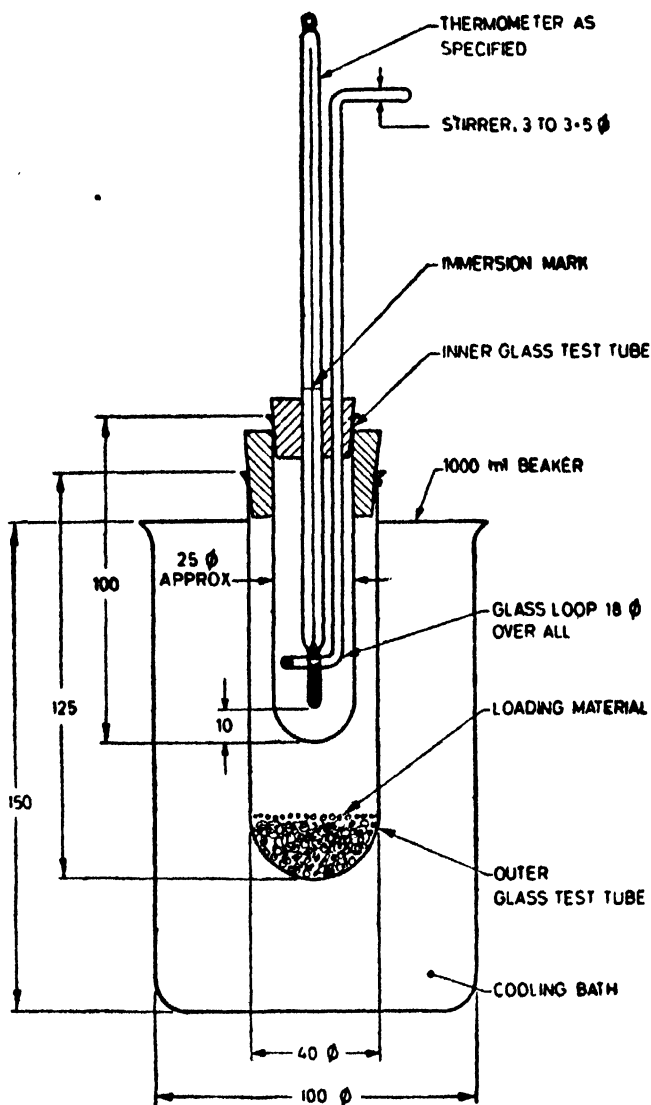
A-1.1.4.1 The thermometer shall bear a certificate from the National Physical Laboratory, New Delhi, or any other institution authorized by the Government of India to issue such a certificate.

A-1.2 Preparation of Sample for Test — Introduce about 150 ml of the material in a dry, wide-mouthed, glass stoppered bottle having 450 ml capacity and stopper the bottle. Then add about 10 g of powdered potassium hydroxide. Keep the whole for two hours with occasional shaking. Later decant and filter through a dry filter paper into the inner tube of the crystallizing point apparatus.

A-1.3 Procedure -- Remove the inner tube of the apparatus from its jacket and introduce a representative portion of the material sufficient to cover the bulb of the thermometer (about 20 ml). Carry out a preliminary rapid cooling of the liquid to determine the approximate crystallizing point. Partially immerse the tube in a bath of about 5°C above the crystallizing point, so that the crystals melt, except for a trace necessary for seeding. Replace the inner tube in its jacket, and assemble the apparatus as shown in Fig. 1, with the cooling bath between 6 to 8°C below the expected crystallizing point. Take thermometer readings at intervals of half a minute, with continuous and gentle stirring. The crystallizing point corresponds to the first five consecutive readings during which the temperature remains constant within 0.1°C.

A-1.3.1 If supercooling takes place, as shown by a rise in temperature, the constant temperature shall be observed immediately after the temperature rise. A temperature rise of 1°C shall be regarded as the maximum allowable.

A-1.3.2 If a constant temperature is not obtained over the first five readings after the rise in temperature, six readings shall be taken commencing with the point at which the maximum temperature is first attained. The readings shall be plotted on graph paper against time



All dimensions in millimetres.

FIG. 1 APPARATUS FOR DETERMINATION OF CRYSTALLIZING POINT

intervals and a straight line drawn to lie evenly between the first and second and between the fifth and sixth of the six points just mentioned. This line shall be produced backwards until it intersects the portion of the curve before the temperature-rise. The point of intersection shall, in this case, be taken as the crystallizing point.

A-2. SOLUBILITY IN HYDROCHLORIC ACID

A-2.1 Weigh accurately about 2 g of the material and add 10 ml of 10 percent (*v/v*) hydrochloric acid. Stir to dissolve completely; warm if necessary. Not more than a slight opalescence shall be observed.

A-3. DETERMINATION OF MOISTURE CONTENT

A-3.1 Determine the moisture content of the material by Karl Fischer method according to IS : 2362 - 1973* or by Dean and Stark method.

A-4. DETERMINATION OF PURITY

A-4.1 Outline of the Method — 3-Chloroaniline is estimated by direct titration with standard sodium nitrite solution using starch iodide paper as indicator.

A-4.2 Reagents

A-4.2.1 Sodium Nitrite Solution — approximately 0.2 N, freshly standardized.

A-4.2.2 Starch Iodide Papers

A-4.2.3 Concentrated Hydrochloric Acid — Conforming to IS : 265-1976†.

A-4.3 Procedure - Accurately weigh by difference from a weighing bottle about 10 g of the material and transfer to a beaker containing 400 ml of water at 60°C and 50 ml of hydrochloric acid, stir to dissolve completely. Cool to 20°C. Make up to 1 000 ml in a volumetric flask.

Transfer by means of a pipette 100 ml of the above solution into a beaker containing 200 g of crushed ice and 200 ml water with 5 g of potassium bromide and 20 ml concentrated hydrochloric acid. While stirring mechanically titrate with sodium nitrite solution, as rapidly as possible to a distinct blue ring test on a starch iodide paper. The end point is taken when the test persists for 5 minutes without further addition of nitrite.

*Determination of water by the Karl Fischer method (*first revision*).

†Specification for hydrochloric acid (*second revision*).

A-4.4 Calculation

$$\text{Purity, percent by mass} = \frac{127.57 \times V \times N}{M}$$

where

V = volume in ml of sodium nitrite solution used,

N = normality of sodium nitrite solution, and

M = mass in g of the material used for the test.

A-5. DETERMINATION OF IMPURITIES

A-5.0 General — The impurities in 3-chloroaniline is determined by ascending thin-layer chromatographic method.

A-5.1 Apparatus

A-5.1.1 *Micropipette*

A-5.1.2 *Developing Chamber*

A-5.1.3 *Thin-layer Chromatographic Glass Plates* — 20×20 cm.

A-5.1.4 *Chromatographic Sprayer*

A-5.1.5 *Adjustable Thin Plate Applicator*

A-5.1.6 *Weighing Bottle*

A-5.1.7 *Oven*

A-5.2 Reagents

A-5.2.1 *Silica Gel G Neutral*

A-5.2.2 *Developing Solvent* — Benzene : Chloroform (95 : 5) (v/v).

A-5.2.3 *Sample Solution* — 1 percent in methanol.

A-5.2.4 *Reference Solutions*

- a) 0.005 percent solution of 100 percent aniline in methanol,
- b) 0.005 percent solution of 100 percent 2-chloroaniline in methanol,
and
- c) 0.005 percent solution of 100 percent 4-chloroaniline in methanol.

A-5.2.5 *Spray Reagent* — N-1-Naphthylethylenediamine dihydrochloride (1 percent in water).

A-5.3 Procedure

A-5.3.1 Preparation of Chromatographic Plates — Mix about 50 g of neutral silica gel G with 85 to 90 ml of water for three minutes to form a uniform slurry then by means of the applicator coat the plate with this slurry to get silica gel G layer of thickness not exceeding 0.25 mm. Activate it in the oven maintained at 110°C for 90 minutes. By means of a needle, mark a thin line widthwise on all plates at about 3 cm from the upper edge. Also remove a band of about 5 mm width silica gel G from either of the lateral sides of each plate.

A-5.3.2 Chromatographic Separation — Pipette 10 μ l of the sample solution (A-5.2.3) and 10 μ l of reference solutions (A-5.2.4) and apply in the form of a uniform spot on the plate, at a distance of 3 cm from the bottom edge. After complete evaporation of the solvent, place the plate in the chromatographic chamber and allow the developing solvent (A-5.2.2) to run up to 1.4 cm at room temperature. Take out the plate and dry completely. Keep the plate for 2 minutes in a chamber containing nitrous acid fumes (prepared by mixing sodium nitrite and hydrochloric acid). Blow out the excess nitrous fumes from the plate and spray with the spraying reagent (A-5.2.5). The material and the constituent impurities are identified by their respective characteristic colours as under:

<i>Substance</i>	<i>Zone</i>	<i>Ref Value</i>	<i>Colour</i>
Aniline	I	0.31	Bluish-violet
4-Chloroaniline	II	0.38	Blue
3-Chloroaniline	III	0.47	Red
2-Chloroaniline	IV	0.67	Brownish-red

A-5.4 Reporting — Report impurity as that which is nearest in intensity to the reference samples. In case the colour intensity does not come in the range of the standard spots of impurity, repeat the whole procedure using suitable concentrations of various impurities.

APPENDIX B

(Clause 4.1)

SAMPLING OF 3-CHLOROANILINE

B-1. GENERAL REQUIREMENTS

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 Samples shall be taken at a place protected from damp air, dust and soot.

B-1.2 Sampling instrument shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container, selected for sampling, shall be mixed as thoroughly as possible, by suitable means.

B-1.5 The samples shall be placed in a clean, dry and air-tight glass-stoppered containers on which the material has no action.

B-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-1.7 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling, year of manufacture, and other important particulars of the consignment.

B-1.8 Samples shall be stored in a cool and dry place.

B-2. SAMPLING INSTRUMENT

B-2.0 The following forms of sampling instruments may be used:

- a) Sampling bottle or can for taking samples from various depths in large tanks; and
- b) Sampling tube.

B-2.1 Sampling Bottle or Can — It consists of a weighed bottle or metal container with removable stopper or top, to which is attached a light chain (*see* Fig. 2). The bottle or can is fastened to a suitable pole. For taking a sample, it is lowered in the tank to the required depth, and the stopper or top is removed by means of the chain for filling the container.

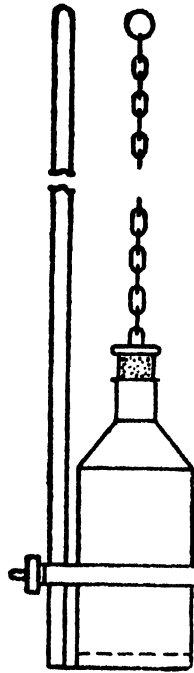


FIG. 2 SAMPLING BOTTLE OR CAN

B-2.2 Sampling Tube— It is made of metal or thick glass and is about 20 to 40 mm in diameter and 400 to 800 mm in length (see Fig. 3). The upper and lower ends are conical and reach 5 to 10 mm diameter at the narrow ends. Handling is facilitated by two rings at the upper end. For taking a sample, the apparatus is first closed at the top with the thumb or a stopper and lowered until the desired depth is reached. It is then opened for a short time to admit the material and finally closed and withdrawn.

B-2.2.1 For small containers, the size of the sampling tube may be altered suitably.

B-3. SCALE OF SAMPLING

B-3.1 Lot — In any consignment all the containers of the same size and drawn from the same batch of manufacture, shall constitute a lot. If a consignment is known to consist of different batches of manufacture or of different sizes of containers, the containers belonging to the same batch and size shall be grouped together and each group shall constitute a separate lot.

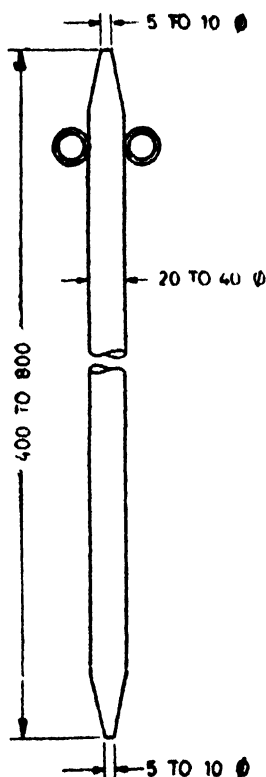


FIG. 3 SAMPLING TUBE

B-3.2 Tests shall be conducted on each lot separately for ascertaining its conformity to the requirements of this specification. The number of containers to be chosen at random from the lot for this purpose shall depend on the size of the lot and shall be in accordance with col 1 and 2 of Table 2.

B-3.3 The containers shall be chosen at random from the lot with the help of a suitable random number table. In case no such table is available, the following procedure is recommended for use:

Starting from any container from the lot, count them as 1, 2, ... , up to r and so on, in one order, where r is the integral part of N/n (N being the number of containers in the lot and n being the number of containers to be selected). Every r th container thus counted shall be withdrawn to give samples for tests.

B-4. PREPARATION OF TEST SAMPLES

B-4.1 From each of the containers selected as in **B 3.2**, a small representative portion of the material from different parts of the container shall be drawn with the help of a suitable sampling instrument (*see B-2*).

B-4.2 Out of these portions a small but equal quantity of material shall be taken and mixed thoroughly to form a composite sample. The composite test sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

TABLE 2 SCALE OF SAMPLING

(*Clause B-3.2*)

LOT SIZE	NO. OF CONTAINERS TO BE SELECTED
(<i>N</i>)	(<i>n</i>)
(1)	(2)
Up to 15	2
16 „ 25	3
26 „ 50	4
51 and above	5

B-4.3 The remaining portion of the material from each of the containers (*see B-4.2*) shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the containers sampled shall be marked for the purchaser, another for the supplier and the third to be used as a referee sample.

B-4.4 All the individual and composite samples shall be transferred to separate containers and shall be sealed and marked with full identification particulars given under **B-1.7**.

B-4.5 The referee test samples consisting of a composite sample and a set of *n* individual samples shall bear the seal of both, the purchaser and the supplier. These shall be kept at a place agreed to between the purchaser and the supplier to be used in case of any dispute between the two.

B-5. NUMBER OF TESTS

B-5.1 Tests for determination of moisture content and assay shall be conducted on each of the individual samples (*see B 4.3*).

B-5.2 Tests for the determination of all other characteristics given in 2.1 and Table 1 shall be conducted on the composite sample (*see* **B-4.2**).

B-6. CRITERIA FOR CONFORMITY

B-6.1 For Individual Samples — The lot shall be declared as conforming to the requirements of purity and moisture content if each of the individual test results satisfy the relevant requirements given in Table 1.

B-6.2 For Composite Samples — For declaring the conformity of a lot to the requirements of all other characteristics tested on the composite sample (*see* **B-4.2**), the test result for each of characteristics shall satisfy the relevant requirements given in 2.1 and Table 1.



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